ORIGINAL MEASUREMENTS: Moeller, T.; Galasyn, V. J. Inorg. Nucl. Chem. 1960, 12, 259-65.
PREPARED BY:
M. Salomon

EXPERIMENTAL VALUES:

The solubility of SmI_3 in $HCON(CH_3)_2$ at $25^{\circ}C$ was reported as

 520.7 g dm^{-3}

and as

0.4666 mol dm⁻³

The solid phase is the solvate $SmI_3.8HCON(CH_3)_2$. The melting point (sealed tube method) of this solvate given as 97.5 - 100.0°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Authors state that solubilities were determined by analysis of aliquots after equilibration at $25 \pm 0.025^{\circ}$ C, and that techniques were generally similar to those described in (1).

The rare earth content was determined by complexometric titration with EDTA at 60°C. Iodide was determined by the Volhard method, and carbon, hydrogen, and nitrogen by usual microanalytical techniques.

REFERENCES:

- Moeller, T.; Cullen, G.W. J. Inorg. Nucl. Chem. 1959, 10, 148.
- Watt, G.W.; Gentile, P.S.; Helvenston, E.P. J. Am. Chem. Soc. <u>1955</u>, 77, 2752.
- Biltz, H.; Biltz, W. Laboratory Methods of Inorganic Chemistry (2nd Edition). John Wiley. N.Y. 1928.
- Leader, G.R.; Gormley, J.F. J. Am. Chem. Soc. 1951, 73, 5731.
- Thomas, A.B.; Rochow, E.G. J. Am. Chem. Soc. <u>1957</u>, 79, 1843.

SOURCE AND PURITY OF MATERIALS:

The initial material was the rare earth oxide of 99.9+% purity. Iodides were prepd by two methods. 1. Acetyl iodide method (2) where the hydrated acetate is treated with acetyl iodide in benzene. Acetyl iodide prepd as in (3). 2. The iodide was prepd by metathesis by reaction of the hydrated SmCl₃ with KI in DMF followed by addition of benzene and distillation of the benzene-water azeotrope.

For both preparations the solvate $\mathrm{SmI}_3.8\mathrm{DMF}$ was recrystallized from DMF by addition of ether.

The solvent, DMF , was prepared as in (4,5), and its electrolytic conductance was $\geq 3.7 \times 10^{-7} \text{ S cm}^{-1} \text{ at } 25^{\circ}\text{C}$.

ESTIMATED ERROR:

Soly: precision around \pm 0.1% (compiler).

Temp: precision \pm 0.025 K (authors).